CHAPTER III
EXPERIMENTAL

3.1 Materials

All chemicals were obtained from commercial sources and used as received. Aluminum oxide, activated, acidic, Brockman I, standard grade was purchased from Aldrich Co., Ltd. (Munich, Germany) with surface area, average pore diameter and average particle size of 155 m^2/g, 58 °A. and 150 meshes, respectively. Benzene was purchased from BDH Co., Ltd. (Poole, England) with a purity of >99.7%. Toluene was purchased from Lab-Scan Co., Ltd. (Samutsakorn, Thailand) with a purity of > 99.5%. Ethylbenzene was purchased from Fluka Co., Ltd. (Steinheim, Switzerland) with a purity of >98.0%. Sodium hydroxide and hydrochloric acid were supplied from Lab-Scan Co., Ltd. (Samutsakorn, Thailand). Triton X-100, a nonionic surfactant, was purchased from Fluka Co., Ltd. (Steinheim, Switzerland). Sodium dodecyl sulfate, a anionic surfactant, (purity > 99 %) was purchased from Carlo Erba Reagent. (Milan, Italy). Deionized water was obtained from Nontri Co., Ltd. (Bangkok, Thailand).

3.2 Experimental

3.2.1 Adsorption of Surfactant on Aluminum Oxide

Batch adsorption of both single and mixed surfactant systems were carried out as follows. SDS stock solutions were diluted with deionized water to form a series of 20 ml solutions with varying surfactant concentrations. The mixture was added to vials containing 0.4 g of aluminum oxide and adjusted to about pH 3.5 by adding NaOH or HCl. The vials were then placed on a shaker and kept at a constant temperature of 30 °C. After equilibrium was reached, the bulk phase was filtered using syringe filter and SDS concentration in the supernatant was measured using total organic carbon analyzer (TOC) (Shimadzu TOC 5000) (Behrends et al., 1995). Simple mass balance was performed to give the amount of adsorbed SDS per gram of aluminum oxide. Similar adsorption experiments were carried out for the

systems using nonionic surfactant, Triton X-100, and mixtures of SDS and Triton X-100 in the molar ratios; 1:1, 1:3 and 3:1, respectively. For Triton X-100, the supernatant was measured by UV-VIS spectrophotometer (Perkin Elmer, Lamda 10) at wavelength 275 nm (Huang and Gu, 1987). For mixed surfactants, both TOC and spectrophotometer were systematically used to determine the concentration of total surfactants and Triton X-100, and thus, SDS concentration could be subsequently determined. Schematic diagram of the adsorption experiments is shown in Figure 3.1.
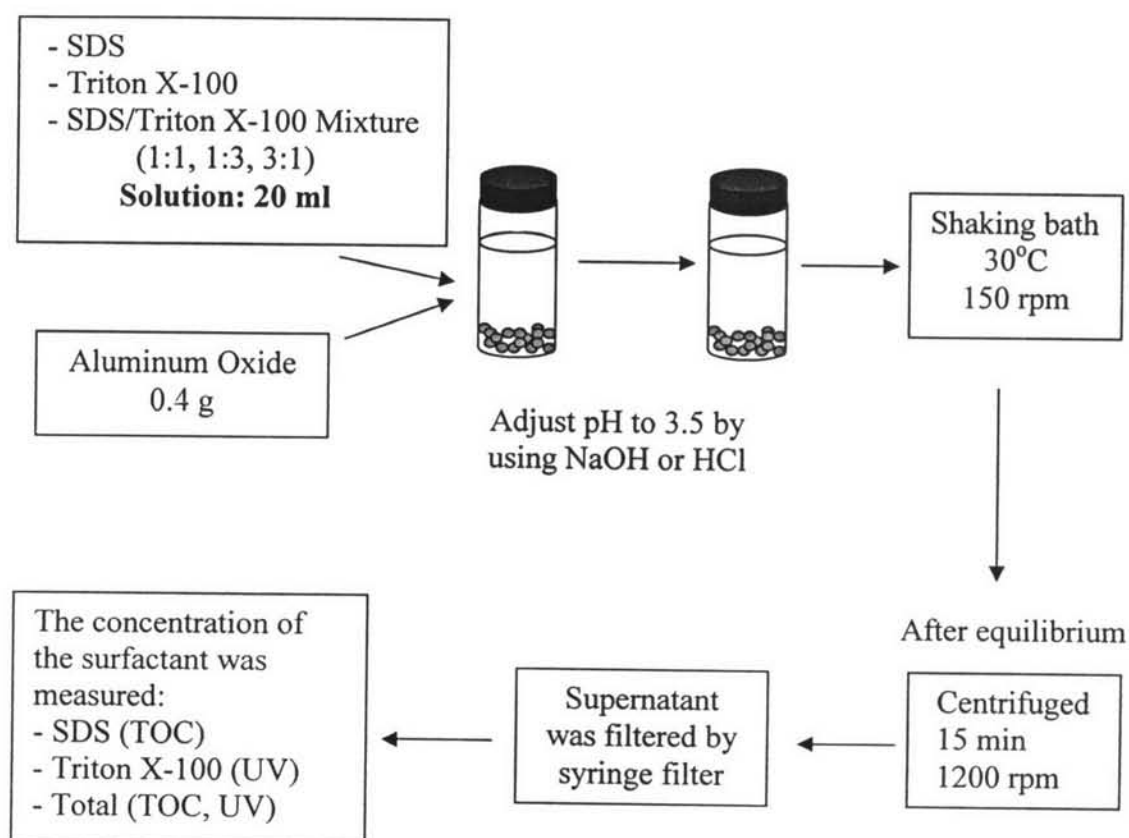

Figure 3.1 Schematic diagram of the adsorbed surfactant on aluminum oxide.

3.2.2 Adsolubilization of Organic Compounds into Surfactant Adsorbed on Aluminum Oxide

The experiments were divided into 2 parts: single surfactant and mixed surfactant systems. For the adsolubilization of single surfactant system (SDS and Triton X-100), the organic compound was added into the equilibrium surfactant

adsorbed on aluminum oxide in the 100 ml glass bottle at pH 3.5. The glass bottle was agitated at ambient temperature until equilibrium for 2 days. After that the solids was separated and the supernatant was then analyzed for organic compounds concentration by a headspace gas chromatography (Nguyen *et al.*, 1988). A simple mass balance was performed to determine the adsolubilized amount of the organic compound. For mixed surfactants systems (SDS/Triton X-100 molar ratios; 1:1, 1:3 and 3:1), the organic compound (benzene, toluene, and ethylbenzene) was added into mixed surfactants equilibrium systems. The experimental conditions and analytical methods were the same as in the single surfactant system. Schematic diagram of the adsolubilization of organic compounds is shown in Figure 3.2.
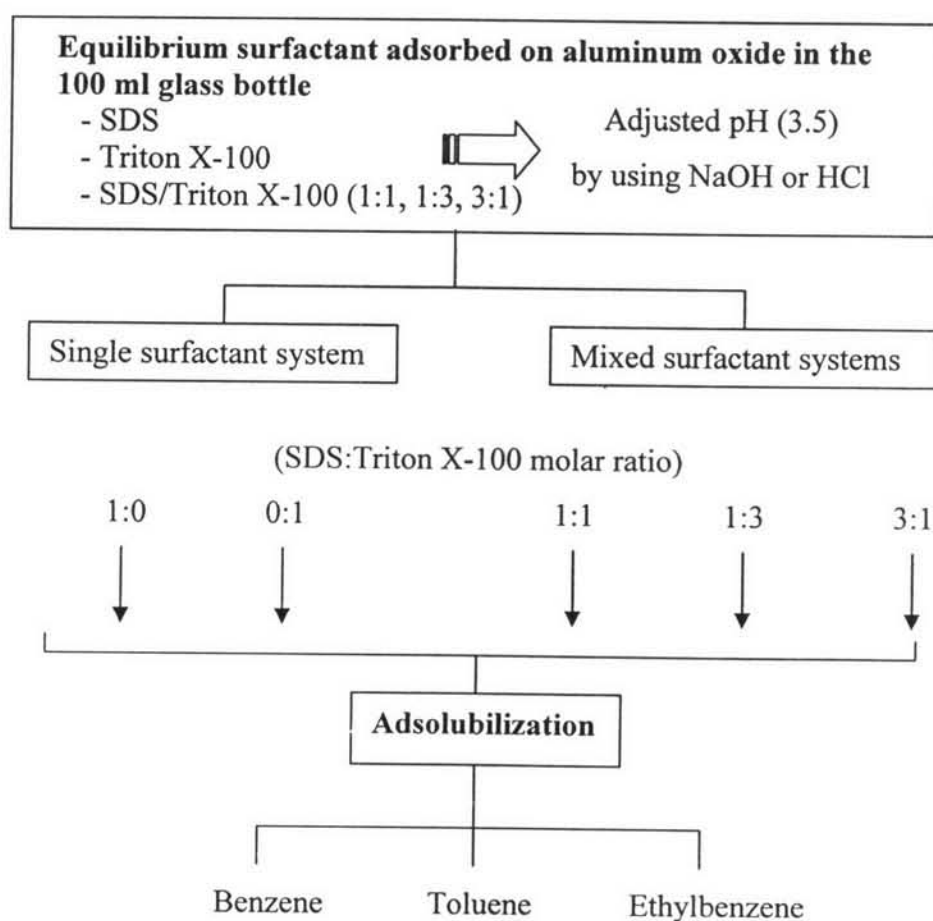

Figure 3.2 Schematic diagram of the adsolubilization of organic compounds.

3.3 Data Analysis

3.3.1 Surfactant Adsorption Isotherm

Adsorption isotherm of SDS, Triton X-100, and mixed surfactants (SDS/Triton X-100 molar ratios; 1:1, 1:3 and 3:1) were constructed by plotting the amount of surfactant adsorbed per gram of aluminum oxide versus equilibrium concentration of surfactant.

3.3.2 Adsolubilization Isotherm

Adsolubilization isotherms of the model organic compounds (benzene, toluene, and ethylbenzene) were generated by plotting the amount of adsolubilized solute versus equilibrium concentration of solute.

3.3.3 Partition Coefficient (K)

The adsolubilization equilibrium constant or partition coefficient (K) was used to describe the distribution of solutes between the aqueous phases and the admicelle pseudophase (Holzheu *et al.*, 2000). K can be determined from the following equation.

$$K = \frac{X_{admicelle}}{X_{bulk}}$$

Where $X_{admicelle}$ =   mole fraction of solute in the surfactant coverage

         X_{bulk} =   mole fraction of solute in the bulk

Partition coefficient plots (K) were generated by plotting the partition coefficient (K) versus mole fraction of solute in the admicelle ($X_{admicelle}$).